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Wear and Friction Behavior of Zr Implanted D3 Steel

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Abstract

Multicharged, pure, high current and pulsed ion beams of Zr have been extracted from a metal vapour vacuum arc (MEVVA) source and implanted into AISI D3 (C: 2-2,35%, Mn: 0,60%, Si: 0,60%, Cr: 11-13,50%, Ni: 0,30%, W: 1%, V: 1%) tool steel samples at the $3,6 \cdot 10^{16}$, $5 \cdot 10^{16}$ and $1 \cdot 10^{17}$ ions/cm² doses. The wear resistance and friction coefficient have been estimated using pin-on-disc wear tests. Implantation of Zr decreased the wear loss and friction coefficient. RBS, AES and SEM Microprobe analyses were used as a guide for explanation of implantation's effects.

Introduction

Ion implantation is a process extensively used for modifying the physical or chemical properties of the near surface regions of a solid (typically 0.01 to 0.5 μm in depths). Atoms are embedded into the material from a beam of energetic ions (2 to 1000 keV), or from a plasma. Ion implantation using ion-beams are carried out in high vacuum (10^{-5} to 10^{-6} Torr) (1). While nitrogen is the most frequently used ion for non-semiconductor applications and has been thoroughly studied, other ions are finding applications especially in those areas or materials where nitrogen does not provide a significant improvement (2).

After the development of MEVVA (metal vapour vacuum arc) source led by Brown and coworkers at Lawrence Berkeley National Laboratory, ion implantation has advanced to include the implantation of various metallic ions for improving not only tribological properties, but also such other surface properties as chemical stability and engineering reliability etc (3,4,5). Extensive work on the implantation of Ti, Cr, Ta, W, Zr, Al, Mo and coimplantation of such ions with C or O on steels has been extensively carried out by groups at LBL in USA, ANSTO in Australia, Dokuz Eylül University in Turkey with the objective to produce an enhance surface properties such as good wear resistance and low friction coefficient (6,7,8).

In this paper, describe our investigation of the effect of Zr ion implantation on the tribological properties of D3 steel. In addition to the wear tests, the surface of the steel is characterized by means of Auger Electron Spectroscopy (AES), Rutherford Backscattering Spectroscopy (RBS) and scanning electron microscopy with a microprobe. The results of the tribological tests are tentatively explained in view of the results of such analyses.

2. Experimental Details

Ion implantation was performed using the broad-beam MEVVA ion source at LBNL, which has been previously described. (9,10). A pulsed Zr ion beam with pulse length of 250 μs and repetition rate of about 2 pulses per second was used. Beam extraction voltage was 50 kV, which corresponds to a mean ion energy of 130 keV since the mean charge state for the Zr ion is 2.6 (11). Pressure in the vacuum chamber was 2×10^{-6} Torr. Beam current was 200 mA, which corresponds to a current density of about 20 mA/cm².

Before implantation, the steel samples were polished to a mirror finish, and ultrasonically cleaned with acetone and ethanol. The samples were then mounted on a water cooled sample holder to prevent excessive heating. Zr⁺ ions were implanted at doses of 3.6×10^{16} , 5×10^{16} and 1.1×10^{17} ions/cm².

Wear measurements were performed on a pin-on-disc tribometer (CSEM) using a Al₂O₃ ball (in 6 mm diameter) in alcohol. It was carried out under a load of 1, 2 and 5 N at a constant speed of 0.02 ms⁻¹ for 1000 laps. The temperature was 22 °C. The friction values were recorded during tests. Wear track profiles were measured with a profilometer (Mahr Perthometer PRK).

In-depth composition profiles were obtained by Rutherford Backscattering Spectroscopy (RBS) using 2 MeV He⁺ beam. Auger Electron Spectroscopy was carried out in a Physical Electronics PHI 660 SAM and the elemental mapping was obtained in a scanning electron microscope equipped with a wavelength dispersive x-ray spectrometer.

3. Results and Discussion

Prior to the implantation, the mean charge state of Zr ions in the beam was determined using a time-of-flight spectrometer. This TOF spectrum was taken 100 μs after pulse initiation, and it is shown in Figure 1. The concentration of the different charged species is proportional to the area under the peaks. The mean energy is calculated from the accelerating voltage and the mean charge state.

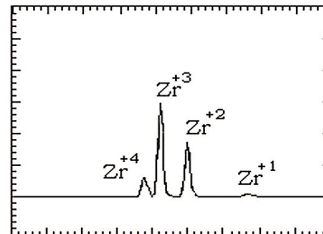
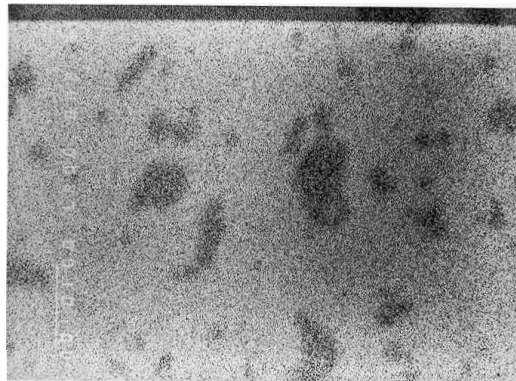
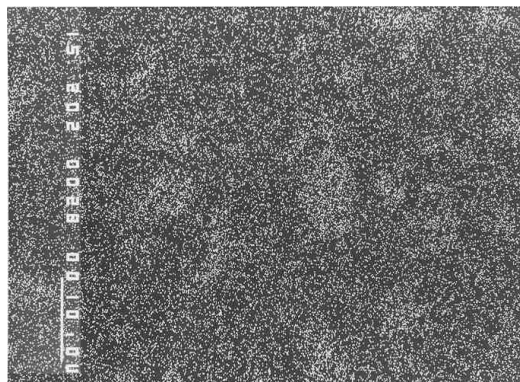


Fig.1. A spectra which was taken during implantation process

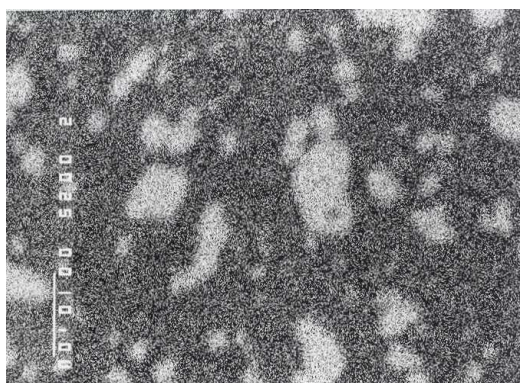
The microstructure of the D3 steel used in this investigation consisted of an Fe-rich matrix with chromium carbide precipitates. This is shown in Figure 2, where the elemental mapping for iron, carbon and chromium are shown. Figure 2 also shows our attempt to carry out the Zr elemental mapping for the sample implanted with a retained dose of $5 \times 10^{16} \text{ cm}^{-2}$. As one can see no preferential accumulation of Zr was detected in the different phases.



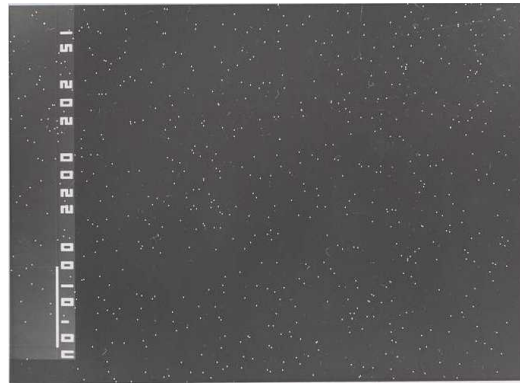
Surface distribution of Fe



Surface distribution of C



Surface distribution of Cr



Surface distribution of Zr

Fig. 2. The SEM microprobe surface analyse images

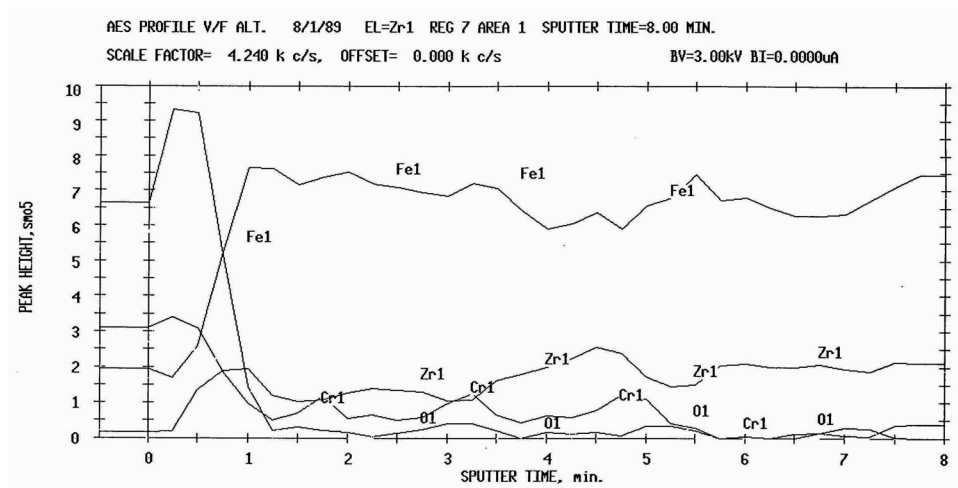


Fig. 3. AES profile

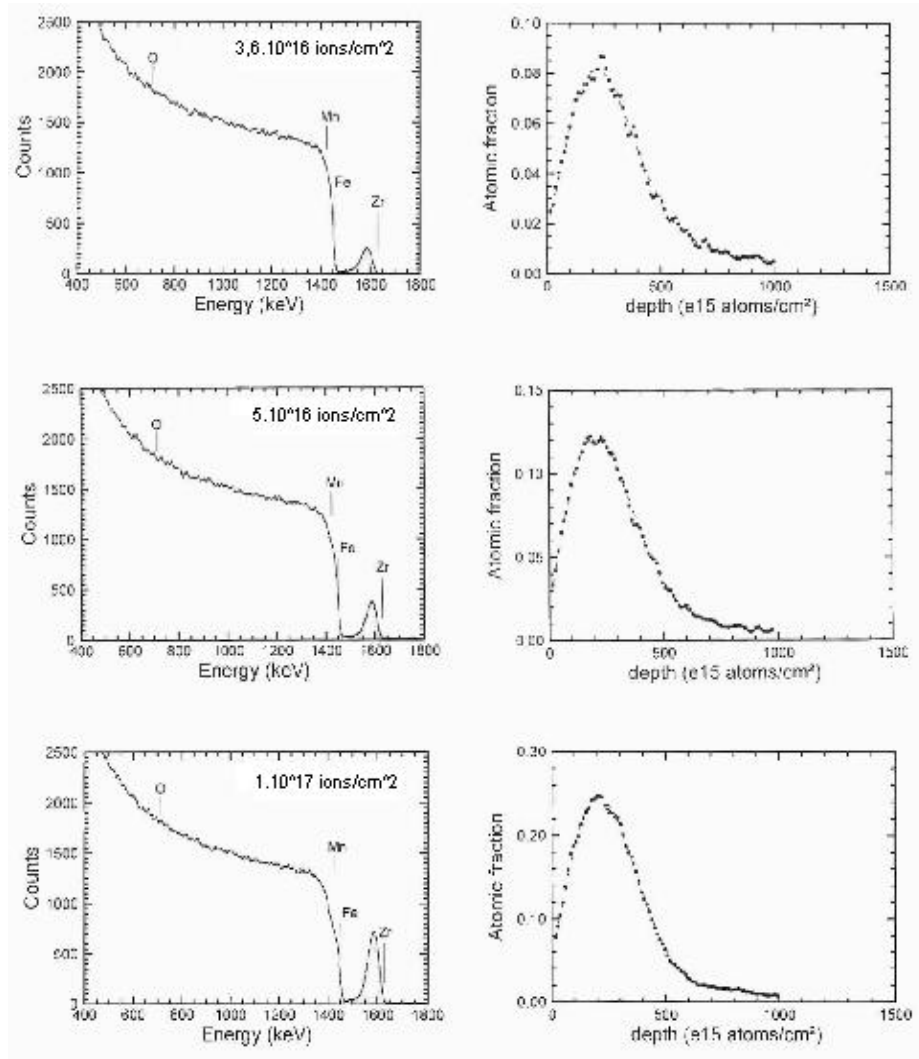


Fig. 4. The RBS results

Other works (12,13) indicated that high-dose implantation can form an amorphous layer embedded with nano-particles.

The AES profile indicates the presence of a thin oxide layer at the surface of the implanted samples. The presence of oxidize layers on steel samples which have been manipulated in openatmosphere is highly epected. Those tend to be chromium rich, which prevents further oxidation. The in-depth AES elemental distribution (Fig 3) supports the finding of Zr in the steel RBS provides a more quantitative evaluation of the amount of retained Zr in the implanted samples. The RBS in-depth Zr distribution is shown in Figure 4.

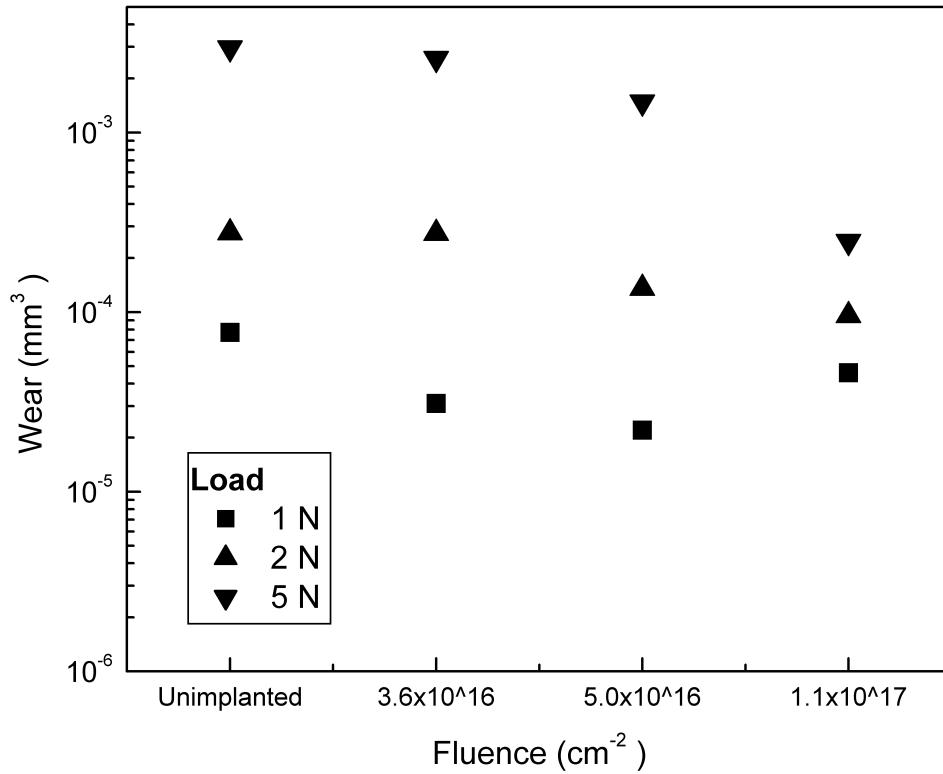


Fig. 5. Wear amount of implanted and unimplanted samples

The wear of the unimplanted samples and the samples implanted with different doses are shown in Figure 5. At the highest dose, 2 N and 5 N, the dependence of wear on fluence is similar, namely wear resistance increases with increasing retained dose. The dependence of wear on the fluence when a 1 N load is applied is more complex. It initially decreases as the fluence increases, and then increases again at the largest retained dose employed. For a load of 1 N, a similar behavior has been recently reported by the authors for the wear in the absence of alcohol. In that case, we have concluded that such a behavior could have resulted to surface fractures due to surface embrittlement resulting to the high dose implantation. This phenomenon can be in principle less important at larger loads because of the larger indentation depths.

The average friction coefficient of the unimplanted and implanted samples are plotted in Figure 6 for the three different loads utilized in this investigation: 1 N, 2 N and 5 N. For doses up to $5 \times 10^{16} \text{ cm}^{-2}$, the change in friction coefficient is within the uncertainty of the measurement. The decay of friction coefficient with increasing load cannot be explained in terms of the presence new phases in the steel, since all our attempts (not explicitly described here) failed to resolve phases in the implanted samples that were not present in the unimplanted ones. It has been suggested that a decrease in friction coefficient with increasing load can result from a stratified structure of the oxides present on the steel surface. Typically steel will oxidize forming an uppermost layer of Fe_2O_3 on top of a Fe_3O_4 on top of a FeO on top of the metal (14). Steels contained high Cr

contents tend to have a Cr_2O_3 protective layer. Zr incorporation into steel will have a somewhat similar effect as Cr: it will oxidize preferentially to Fe, favoring the stratification hypothesis. The Auger results seen in Figure 3 indicate that such stratification in fact existed, and the oxide surface is richer in Cr than the underlying steel. The large decrease in friction coefficient observed in the samples with highest dose is not clearly understood. A correlation between friction coefficient and wear rate fails to explain the results observed here.

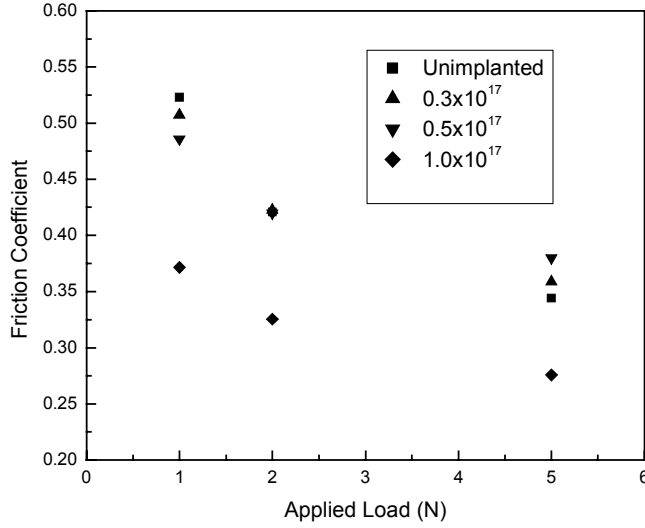


Figure 6: Average friction coefficient measured at the different loads for the unimplanted and implanted samples.

4. Summary and Conclusions

We have investigated the change in tribological properties of D3 steel when implanted with Zr ions with different doses. Even at the highest doses, namely 1×10^{17} , no evidence of formation of new phases was observed. At the implantation conditions used here Zr content peaked at about 20 – 30 nm. A noticeable increase in wear resistance was observed with increasing dose for the large applied loads (2 and 5 N), whereas for 1 N this is not the case. A large reduction in friction coefficient was observed at the highest dose, but it remained virtually unchanged for the lower doses.

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